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Crystal structure of (4*S*,4*aS*,6*aR*,6*bR*,12*aS*,12*bR*,14*aS*,14*bR*)-3,3,6*a*,6*b*,9,9,12*a*-heptamethyloctadecahydro-1*H*,3*H*-4,14*b*-ethanophenanthro[1,2-*h*]isochromene, C₃₀H₅₀O

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Abstract

C₃₀H₅₀O, orthorhombic, *P*₂₁₂₁₂₁ (no. 19), *a* = 6.7397(7) Å, *b* = 12.0404(12) Å, *c* = 30.864(3) Å, *V* = 2504.6(4) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0555, *wR*_{ref}(*F*²) = 0.1234, *T* = 293(2) K.

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Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The air-dried and powdered plants of *Pyrrosia lingua* were extracted with 90% EtOH under reflux three times for 1 h each time. The combined extracts were concentrated and suspended in H₂O. The suspension was extracted with AcOEt. The title compound (130 mg) was isolated from AcOEt by the silica gel column chromatography with petroleum ether/AcOEt (9:1) and then the sephadex LH-20 with dichloromethane/methanol (volume ratio 1:1). Recrystallisation from AcOEt gave colorless needle crystals of the title compound within 2 days [5].

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.23 × 0.22 × 0.21 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.07 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ _{max} , completeness:	25.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	23641, 4411, 0.067
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3217
<i>N</i> (<i>param</i>) _{refined} :	282
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.1816(6)	−0.0134(3)	0.23814(11)	0.0425(9)
H1A	0.2603	0.0338	0.2569	0.051*
H1B	0.0597	0.0258	0.2314	0.051*
C2	0.1306(6)	−0.1197(3)	0.26236(12)	0.0502(10)
H2A	0.2522	−0.1553	0.2719	0.060*
H2B	0.0535	−0.1013	0.2879	0.060*
C3	0.0136(6)	−0.2000(3)	0.23435(12)	0.0489(10)
H3A	−0.1160	−0.1683	0.2286	0.059*
H3B	−0.0063	−0.2684	0.2504	0.059*
C4	0.1138(6)	−0.2279(3)	0.19088(12)	0.0414(9)
C5	0.1778(5)	−0.1186(3)	0.16841(10)	0.0367(8)
H5	0.0526	−0.0805	0.1621	0.044*
C6	0.2730(6)	−0.1346(3)	0.12427(11)	0.0447(10)
H6A	0.2012	−0.1913	0.1084	0.054*
H6B	0.4086	−0.1598	0.1280	0.054*
C7	0.2720(7)	−0.0266(3)	0.09826(11)	0.0454(10)
H7A	0.1356	−0.0058	0.0923	0.055*
H7B	0.3370	−0.0395	0.0707	0.055*
C8	0.3760(5)	0.0710(3)	0.12135(10)	0.0333(8)
C9	0.3030(5)	0.0774(3)	0.16943(10)	0.0333(8)
H9	0.1636	0.0998	0.1672	0.040*
C10	0.2972(5)	−0.0337(3)	0.19571(11)	0.0341(8)
C11	0.4038(6)	0.1744(3)	0.19300(11)	0.0391(9)
H11A	0.3502	0.1800	0.2221	0.047*
H11B	0.5447	0.1592	0.1954	0.047*
C12	0.3748(6)	0.2847(3)	0.16975(10)	0.0383(9)
H12A	0.4502	0.3419	0.1845	0.046*
H12B	0.2357	0.3053	0.1708	0.046*
C13	0.4416(5)	0.2783(3)	0.12259(10)	0.0319(8)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H13	0.5783	0.2508	0.1240	0.038*
C14	0.3254(5)	0.1855(3)	0.09816(10)	0.0324(8)
C15	0.3921(6)	0.1839(3)	0.05014(10)	0.0424(9)
H15A	0.5270	0.1561	0.0487	0.051*
H15B	0.3081	0.1326	0.0343	0.051*
C16	0.3838(6)	0.2973(3)	0.02800(11)	0.0435(9)
H16A	0.4274	0.2908	−0.0018	0.052*
H16B	0.2487	0.3252	0.0281	0.052*
C17	0.5183(5)	0.3763(3)	0.05247(10)	0.0372(9)
H17	0.6476	0.3392	0.0535	0.045*
C18	0.4580(5)	0.3922(3)	0.09980(10)	0.0333(8)
C19	0.6318(6)	0.4631(3)	0.11673(12)	0.0479(10)
H19A	0.7350	0.4163	0.1288	0.057*
H19B	0.5867	0.5143	0.1389	0.057*
C20	0.5641(5)	0.4972(3)	0.03979(12)	0.0423(9)
H20	0.6309	0.5004	0.0116	0.051*
C21	0.7093(6)	0.5268(3)	0.07681(13)	0.0557(11)
H21A	0.8431	0.5037	0.0696	0.067*
H21B	0.7092	0.6062	0.0822	0.067*
C22	0.3825(6)	0.5732(3)	0.04048(11)	0.0451(10)
C23	−0.0407(7)	−0.2878(3)	0.16285(14)	0.0613(12)
H23A	0.0170	−0.3065	0.1354	0.092*
H23B	−0.0831	−0.3544	0.1772	0.092*
H23C	−0.1529	−0.2400	0.1584	0.092*
C24	0.2850(7)	−0.3109(3)	0.19798(14)	0.0588(12)
H24A	0.3859	−0.2768	0.2155	0.088*
H24B	0.2355	−0.3758	0.2124	0.088*
H24C	0.3403	−0.3316	0.1705	0.088*
C25	0.5067(6)	−0.0729(3)	0.20848(12)	0.0480(10)
H25A	0.5866	−0.0097	0.2160	0.072*
H25B	0.5662	−0.1113	0.1845	0.072*
H25C	0.4980	−0.1220	0.2329	0.072*
C26	0.6023(6)	0.0458(3)	0.11913(12)	0.0478(10)
H26A	0.6236	−0.0327	0.1223	0.072*
H26B	0.6693	0.0845	0.1421	0.072*
H26C	0.6537	0.0699	0.0917	0.072*
C27	0.0992(5)	0.2068(3)	0.09865(11)	0.0408(9)
H27A	0.0528	0.2083	0.1281	0.061*
H27B	0.0714	0.2768	0.0850	0.061*
H27C	0.0328	0.1484	0.0832	0.061*
C28	0.2705(6)	0.4638(3)	0.10118(12)	0.0388(9)
H28A	0.1661	0.4269	0.0850	0.047*
H28B	0.2267	0.4707	0.1310	0.047*
C29	0.2245(7)	0.5441(3)	0.00659(13)	0.0632(13)
H29A	0.2830	0.5452	−0.0218	0.095*
H29B	0.1726	0.4713	0.0124	0.095*
H29C	0.1189	0.5975	0.0079	0.095*
C30	0.4400(7)	0.6939(3)	0.03282(13)	0.0614(12)
H30A	0.4967	0.7015	0.0044	0.092*
H30B	0.3242	0.7400	0.0350	0.092*
H30C	0.5355	0.7164	0.0542	0.092*
O1	0.3003(4)	0.5723(2)	0.08356(8)	0.0464(7)

Experimental details

All hydrogen atoms were positioned geometrically, with the $d(C-H) = 0.96-0.98 \text{ \AA}$, $U_{iso}(H) = 1.2$ times $U_{eq}(C)$ and $U_{iso}(H) = 1.5$ times $U_{eq}(O)$.

Comment

Pyrrosia lingua is documented in the Chinese Pharmacopoeia for the treatment of swelling, urocytitis, urinary calculus, bloody urine, cough, bronchitis, etc. and has also been used to cure infection and diarrhoea in some regions of Guizhou Province, China. It is a prolific source of natural hopane triterpenoids which exhibit cytotoxicity and antiproliferative activity [6, 7].

The bond lengths and angles derived from the title structure are within normal ranges. The title compound contains one five-membered ring which shows an envelope conformation at C17 and five six-membered rings which all present chair conformations (*cf.* the figure). At the same time, the title compound contains an ether bond and seven methyl groups, of which C24, C25, C26, C30 are in β -bond, and C23, C27, C29 are in α -bond. The oxo-bridge was confirmed by the distances $d(C20-O3) = 1.441(3)$, $d(C28-O1) = 1.430(3)$. The intermolecular connection was achieved by van der Waals forces.

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